भारतीय मानक

Indian Standard

IS 17982 : 2022

मिथाइल मेथाक्रिलेट — विशिष्टि

Methyl Methacrylate — Specification

ICS 71.080.70

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

October 2022 **Price Group 7**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Methyl methacrylate (MMA) is an organic compound with the formula $CH_2=C(CH_3)COOCH_3$. It is volatile colourless liquid with a characteristic acrid odour and is a monomer produced on a large scale for the production of poly(methyl methacrylate) (PMMA).

Methyl methacrylate is also used to produce the co-polymer methyl methacrylate-butadiene-styrene (MBS), used as a modifier for PVC. Another application is as cement used in total hip replacements as well as total knee replacements. The chemical may polymerize exothermically if heated or kept in sunlight or contaminated with strong acid or base. If the polymerization takes place inside a container, the container may rupture violently. An inhibitor such as hydroquinone, hydroquinone methyl ester and dimethyl-*t*-butylphenol is added to keep the chemical from initiating polymerization.

Based on the recommendations of Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizer, the Committee has decided to formulate Indian Standard on Methyl methacrylate (MMA).

The composition of the Committee, responsible for the formulation of this standard is given at Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHYL METHACRYLATE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, the methods of sampling and testing for Methyl Methacrylate (MMA).

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most editions of the standards indicated below.

IS No.	*Title*
915 : 2012/ISO 1042 : 1998	Laboratory glassware — One-mark volumetric flasks *(third revision)*
1070 : 1992	Reagent grade water — Specification (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
1448 (Part 16) : 2014/ISO 3675 : 1998	Methods of test for petroleum and its products: Part 16 Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (*fourth revision*)
2362 : 1993	Determination of water by karl fischer method — Test method (*second revision*)
2631 : 2020	Iso propyl alcohol — Specification (*second revision*)
4905 : 2015 /ISO 24153: 2009	Random sampling and randomization procedures (*first revision*)
8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*)

3 REQUIREMENTS

3.1 Description

The material shall be clear colourless liquid. It shall be free from visible impurities and from dust.

3.2 The material shall also comply with the requirements specified in Table 1, when tested according to the methods prescribed in col 4 and col 5 of the Table 1.

Table 1 Requirements for Methyl Methacrylate (MMA)

(*Clauses* 3.2, 5.3 *and* 6.1)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Color, Pt-Co, *Max*	5	—	8768
ii)	Water content, ppm, *Max*	500	—	2362
iii)	Total Acidity, ppm, *Max*	35	A	—
iv)	Inhibitor content, ppm	25 – 50 1)	B	—
v)	Purity, percent by mass, *Min*	99.9	C	—
vi)	Specific gravity at 20°C	0.942 – 0.946	—	1448 (Part 16)

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1) As agreed between buyer and supplier.

4 PACKING AND MARKING

4.1 Packing

The material shall be stored and transported in containers made of steel or high-density polyethylene (HDPE) drums as agreed to between the purchaser and the supplier. Stainless steel drums or bulk containers of various constructions (including ultraviolet radiation protection) may also be used and treated similarly to drums.

4.2 Marking

4.2.1 Each container shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Name of manufacturer and his recognized trade-mark, if any;

c) Net weight;

d) Month and year of manufacture

e) Batch or code number; and

f) Any other statutory requirements.

4.2.2 The containers shall be suitably marked in letters as 'DANGER' and shall be labelled as shown in Fig. 5 of IS 1260 (Part 1). The label shall also bear the following clear and legible instruction:

'STORE IN A WELL-VENTILATED PLACE AWAY FROM HEAT, SPARKS AND FLAME. AVOID CONTACT AS CAUSES SKIN IRRITATION'

4.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in Annex D.

5.2 Number of Tests

Tests for the determination of all the characteristics specified shall be conducted on the composite sample.

5.3 Criteria for Conformity

The material shall be taken as conforming to this specification, if the test results of the composite test sample satisfy the corresponding requirements given in **3** and Table 1.

6 TEST METHODS

6.1 Test shall be conducted as prescribed in col 4 and col 5 of Table 1.

6.2 Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070), shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* iii)]

DETERMINATION OF ACIDITY BY ALKALIMETRIC TITRATION

A-1 GENERAL

The sample is mixed with either an equal volume of water or an equal volume of alcohol and is titrated with aqueous sodium hydroxide solution to the phenolphthalein end point.

A-2 APPARATUS

A-2.1 Burette, 10 ml, graduated in 0.05 ml subdivisions.

A-2.2 Erlenmeyer Flask, 250 ml capacity.

A-3 REAGENTS

A-3.1 Alcohols, refined, ethyl alcohol or isopropyl alcohol (*see* IS 2631).

A-3.2 Phenolphthalein Indicator Solution, 10 g/l

Dissolve 1 g of phenolphthalein in ethyl or isopropyl alcohol and dilute to 100 ml with the alcohol.

A-3.3 Sodium Hydroxide, 0.05 N

Prepare and standardize a 0.05 N sodium hydroxide (NaOH) solution.

A-4 PROCEDURE

A-4.1 Measure 50 ml of water into 250 ml erlenmeyer flask, if the sample is completely water-soluble or 50 ml of alcohol, if the sample is not completely water-soluble. Add 0.5 ml of phenolphthalein indicator. Titrate the water or alcohol with 0.05 N sodium hydroxide solution to the first perceptible pink colour.

A-4.2 Pipette out 50 ml of the sample and add it into the flask containing water or alcohol. Titrate it with 0.05 N sodium hydroxide solution to the same first perceptible pink colour originally obtained.

A-5 CALCULATION

Acid Value (*as* acetic acid), ppm =

$$\frac{V \text{ x } N \text{ x } 60 \text{ x } 1000}{sample\ volume \text{ x } D}$$

where

V = sodium hydroxide solution required for titration of the sample, ml;

N = normality of the sodium hydroxide (NaOH) solution; and

D = density of specimen, g/ml.

ANNEX B

[*Table* 1, *Sl No.* iv)]

DETERMINATION OF INHIBITOR CONTENT

B-1 GENERAL

A sample is injected on a gas chromatographic capillary column and the results are calculated using the external standard method with area normalization.

B-2 APPARATUS

B-2.1 Gas Chromatograph

Any gas chromatograph equipped with a flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition.

B-2.1.1 *Column*

Capillary column with (5 percent-phenyl)-methylpolysiloxane non polar phase, with length 30 m; internal diameter 0.32 mm and film thickness 0.25 µm.

B-2.1.2 *Gas chromatograph settings*

B-2.1.2.1 *Injector condition*

Temperature	250°C
Split ratio	20 : 1
Split flow	40 ml/min
Initial pressure	96.28 kPa (13.96 psi) approximately
Mode	constant pressure
Column flow	2.2 ml/min
Average velocity	0.4345 m/sec
Carrier gas	Nitrogen
Injector volume	1 µl

B-2.1.2.2 *Column oven temperature programming*

Heating Rate, °C/min	*Temperature, °C*	*Hold Time, min*
—	150	6
20	250	2.5

B-2.1.2.3 *Detector condition*

Detector type	Flame Ionization Detector (FID)
Temperature	250°C
Hydrogen flow	30 ml/min
Air flow	300 ml/min
Mode	constant makeup
Make up flow	20 ml/min

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packe /capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard

B-2.2 Volumetric flasks (*see* IS 915).

B-2.3 Analytical balance, with least count of 0.1 mg

B-3 REAGENTS

Unless otherwise specified, AR grade chemicals shall be used.

B-3.1 2,4-Dimethyl-6-*tert*-butylphenol (Topanol A)

B-3.2 Methyl Methacrylate (MMA)

B-4 STOCK AND STANDARD SOLUTION PREPARATION

B-4.1 Preparation of 1 000 ppm stock solution

Take about 0.1 g of topanol A in 100 ml volumetric flask and dilute up to the mark with methyl methacrylate (MMA).

B-4.2 Preparation of Standard Solutions

To prepare 10 ppm; 20 ppm; 30 ppm; 50 ppm and 100 ppm standard solutions, take 1.0 ml; 2.0 ml; 3.0 ml; 5.0 ml and 10 ml, respectively, of stock in 100 ml volumetric flask and dilute it up to the mark with methyl methacrylate (MMA).

B-5 SAMPLE PREPARATION

Take approximately 2 ml sample in vial for GC auto sampler or 1 µl for manual injection.

B-6 CALCULATION

B-6.1 Make calibration curve against mg of topanol A content verses area of topanol A.

B-6.2 Concentration of inhibitor can be calculated manually from the calibration curve or automatically from the GC software.

ANNEX C

[*Table* 1, *Sl No.* v)]

DETERMINATION OF METHYL METHACRYLATE CONTENT (PURITY) BY GAS CHROMATOGRAPHY

C-1 GENERAL

A sample and standard are injected on a gas chromatographic capillary column and the results are calculated using the external standard method with area normalization.

C-2 APPARATUS

C-2.1 Gas Chromatograph

Any gas chromatograph equipped with a flame ionization detector (FID), a split/splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition.

C-2.1.1 *Column*

Capillary column with (5 percent-phenyl)-methylpolysiloxane nonpolar phase with length 30 m; internal diameter 320 mm and film thickness 0.25 µm

C-2.1.2 *Gas chromatograph settings*

C-2.1.2.1 *Injector condition*

Temperature	250°C
Split ratio	20 : 1
Split flow	40 ml/min
Initial pressure	96.28 kPa (13.96 psi) approx.
Mode	constant pressure
Column flow	2.2 ml/min
Average velocity	0.4345 m/sec
Carrier gas	Nitrogen
Injector Volume	1 µl

C-2.1.2.2 *Column oven temperature programming*

Heating Rate, °C/min	*Temperature, °C*	*Hold Time, min*
—	150	6
20	250	2.5

C-2.1.2.3 *Detector condition*

Detector type	Flame Ionization Detector (FID)
Temperature	250°C
Hydrogen flow	30 ml/min
Air flow	300 ml/min
Mode	constant makeup
Make up flow	20 ml/min

C-2.2 Syringe, for gas chromatography, 10 µl.

C-2.3 Analytical Balance, with least count of 0.1 mg

C-3 REGENT

C-3.1 Methyl Methacrylate (MMA), AR grade

C-4 RETENTION TIME

Methyl methacrylate (MMA) : 10.5 min

Methacrylic acid : 12.4 min

C-5 CALCULATION

C-5.1 Note the area of both standard and sample.

C-5.2 Purity of the sample =

$$\frac{\text{Area of sample}}{\text{Area of standard x concentration of standard}}$$

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed / capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

ANNEX D

(*Clause* 5.1)

SAMPLING OF METHYL METHACRYLATE

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

D-1.2 Samples shall be taken in a protected area with good ventilation.

D-1.3 Sampling instrument shall be clean and dry.

D-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling, circulation of storage tank or by any other suitable means.

D-1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action. The containers shall be of such size that they are almost completely filled by the sample.

D-1.7 Each sample container shall be sealed air-tight after filling and marked with the manufacturer's name or trademark, the month and year of manufacture of the material, the batch number, and other details of sampling, such as the date of sampling, sampler's name, etc.

D-2 SAMPLING INSTRUMENT

D-2.1 Sampling tube is made of thick glass or metal on which the material has no action and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 1). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

D-2.2 For small containers, the size and shape of the sampling tube may be altered suitably.

D-2.3 In case of sampling from bulk storage tank (through circulation pump sampling valve) suitable sampling container can be used.

D-2.4 In case of filled tanker deeper sampler may be used (sampling from top side) or through sample point arranged to filling line, if tanker/container is under nitrogen pressure (special case)

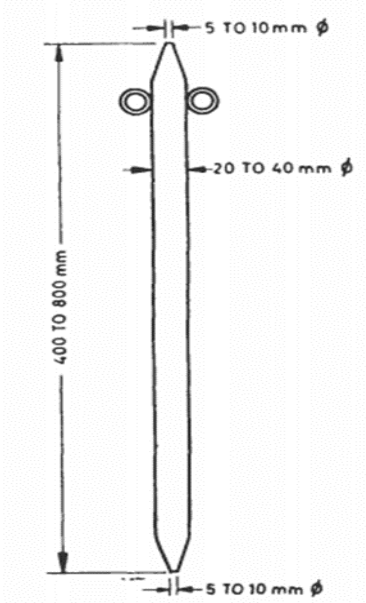

FIG. 1 SAMPLING TUBE

D-3 SCALE OF SAMPLING

D-3.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute a separate lot. In the case of a consignment drawn from a continuous process, 2 000 containers (or 100 metric tons of the material) shall constitute a lot.

D-3.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall he carried out for each lot separately. The number of containers to be chosen from a lot shall depend on the size of the lot and shall be in accordance with co1 1 and 2 of Table 2.

Table 2 Scale of Sampling
(*Clause* D-3.2)

Lot Size *N* (1)	Number or containers to be selected *n* (2)
Up to 3	*
4 to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 to 800	9
801 and above	10

*When the lot size is 3 or less, the number of containers to be selected and the method of judging the conformity of the lot to the specification requirements shall be as agreed between the purchaser and the supplier.

D-3.3 These containers shall be chosen at random from the lot and in order to ensure randomness of selection, random sampling procedures given in IS 4905 may be followed. In case a random number tables are not available; the following procedure may be used:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, …. up to r and so on, where r is the integral part of *N/n* (*N* being the lot size and *n* the number of containers to be selected). Every r^{th} container thus counted shall be withdrawn to give a sample for tests

D-3.4 For continuous production facilities, samples shall be collected from conveyor belt periodically and combined in equal proportion so as to constitute sample representing a lot of respective production capacity.

D-4 PREPARATION OF TEST SAMPLES

D-4.1 From each of the containers selected according to **D-3.3**, a small representative portion of the material sufficient for carrying out tests as indicated in **3** shall be drawn with the help of the sampling tube (*see* **D-2.1**).

D-4.2 Out of these portions, equal quantity of material shall be taken and mixed thoroughly to form a composite sample, not less than 1500 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample

D-4.3 The composite samples shall be transferred to containers of 600 ml capacity and shall be sealed and marked with full identification particulars given in **D-1.7**

D-4.4 The referee test sample shall also bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL, Pune	DR C. V. RODE **(*Chairman*)**
Alkyl Amines Chemicals Limited, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (*Alternate*)
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Jubilant Life Sciences Limited, Noida	SHRI HARI MOHAN LOHANI

Organization	*Representative(s)*
Laxmi Organic Industries, Mumbai	Shri J. P. Suryavanshi Dr. Vijay S. Mishra (*Alternate*)
Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)
National Chemical Laboratory (NCL), Pune	Dr Ravindar Kontham Dr Udaya Kiran Marelli (*Alternate*)
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: PCD 09 (18757).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
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Published by BIS, New Delhi